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(54) Title: ORIENTED POLYMERIC FILM COMPRISING AT LEAST ONE COPOLYETHERESTER

(57) Abstract: An oriented polymeric film comprising one or more copolyetherester(s) wherein the film is oriented in at least one direction; and a waterproof moisture vapour permeable fabric comprising a substrate, for instance a woven or knit material comprising polyester or nylon, the oriented polymeric film, and optionally an adhesive or primer layer or coating between the substrate and the copolyetherester-containing oriented film.

differential transfer of moisture vapour to prevent build-up of moisture. These bicomponent films behave like a permeability valve.

One disadvantage with known waterproof moisture vapour permeable fabrics, such as GORETEX®, is that they are not stretchable. It would be desirable to impart some stretchability to materials of this type while at the same time ensuring that the fabric also has the characteristic of elasticity or stretch recovery, i.e. when the material is stretched it returns to its original dimensions.

SUMMARY OF INVENTION

Oriented polymeric film comprising one or more copolyetherester(s), wherein the film is oriented in at least one direction. Fabric comprising such oriented polymeric film and substrate. Processes for making such oriented polymeric film and fabric.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

A preferred oriented film in accordance with the present invention is a waterproof moisture vapour permeable oriented film and fabric having good mechanical properties (such as toughness, tensile strength, flex performance and tear resistance), preferably having good elasticity or stretch recovery, and preferably having good thermal properties.

In a preferred embodiment, the film has a thickness of less than 50 μm , and preferably less than 20 μm . In a further preferred embodiment the film is a clear film.

Preferred oriented films may also retain an MVTR comparable to a cast film comprising one or more copolyetherester(s). Generally, orientation of a film would be expected to reduce the MVTR of the film in relation to an unoriented film.

The MVTR of the film is primarily dependent upon its chemical composition and thickness, and these parameters can be adjusted to tailor a film so that it is suitable for a particular end-use, as required. In a preferred embodiment of the invention, the MVTR of the copolyetherester is at least about 2500, preferably at least about 3500, and more

The stretched film may be, and preferably is, dimensionally stabilised by heat-setting under dimensional restraint at a temperature above the glass transition temperature of the polymer but below the melting temperature thereof, to induce crystallisation. In
5 applications where film shrinkage is not of significant concern, the film may be heat set at relatively low temperatures or not at all. On the other hand, as the temperature at which the film is heat set is increased, the tear resistance of the film may change. Thus, the actual heat set temperature and time will vary depending on the composition of the film and its intended application but preferably should not be selected so as to substantially degrade the
10 tear resistant properties of the film. Within these constraints, a heat set temperature of about 135° to 205°C is generally desirable.

According to a further aspect of the invention, there is provided a process for the preparation of an oriented film comprising one or more copolyetherester(s), said process
15 preferably comprising the steps of extruding the copolyetherester(s), quenching the extrudate, stretching the quenched extrudate and optionally heat-setting the oriented film.

The film contains preferably at least 50 weight percent, more preferably at least 65 weight percent, more preferably at least 80 weight percent, more preferably at least 90 weight
20 percent, and even more preferably at least 99 weight percent of the copolyetherester(s). The copolyetherester(s) are generally hydrophilic, as described in more detail below.

The viscosity of the copolyetheresters is preferably less than about 3000 Pa.s and preferably at least 20 Pa.s, measured according to the standard ISO11443. Preferably, the
25 viscosity is in the range from about 20 to about 2000 Pa.s, more preferably from about 40 to about 1000 Pa.s, and more preferably from about 50 to about 700 Pa.s, measured according to the standard ISO11443. The viscosity in Pa.s is measured according to the standard ISO 11443 as a function of shear rate in sec^{-1} and temperature. The temperatures used in the measurement of viscosity are from a minimum of just above the melting (or
30 softening) point of the polymer (typically from about 200 to about 210°C) up to a maximum of just above the temperatures (typically from about 230 to about 260°C, particularly from about 240 to about 250°C) used in the processing methods (for example, coextrusion, injection moulding and lamination) of thermoplastic materials. The

In a preferred embodiment, the fabric comprises an adhesive or primer layer or coating between the substrate and copolyetherester-containing film. The adhesive or primer may be any conventional adhesive known in the art, such as a polyurethane-based adhesive. A suitable adhesive is LIOFOL® (UK4501; Henkel). The adhesive or primer is generally applied to the substrate layer. The adhesive or primer layer may be continuous or discontinuous, but should not significantly affect the breathability properties of the fabric. Preferably the adhesive layer is a discontinuous layer.

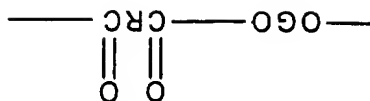
The fabric of the invention should preferably display good durability after washing, i.e. the fabric should retain good structural integrity after repeated washing.

Formation of the fabric according to the invention may be effected by conventional techniques well-known in the art. In a preferred embodiment, the fabric is formed using conventional laminating techniques, for example by lamination of a preformed copolyetherester-containing layer with the substrate, or by casting. Typically, such lamination techniques would involve thermal lamination of the respective layers on hot roll calendaring equipment, and with the application of sufficient pressure, the layers become bonded.

20 Preferred copolyetherester(s) are now described.

In a preferred embodiment of the invention, the copolyetherester elastomer(s) are selected from those described in US Patent No 4,725,481, the disclosure of which is incorporated herein by reference.

25 In another preferred embodiment, the copolyetherester elastomer(s) have a multiplicity of recurring long-chain ester units and short-chain ester units joined head-to-tail through ester linkages, said long-chain ester units being represented by the formula:



(I)

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The poly(alkylene oxide)glycols used to make the copolyetheresters should preferably contain ethylene oxide groups in amounts that result in a copolyetherester having from about 20 to about 68, preferably from about 25 to about 68, more preferably from about 30 to about 55, weight percent ethylene oxide groups, based on the total weight of the copolyetherester. The ethylene oxide groups cause the polymer to have the characteristic of being readily permeable to moisture vapour and, generally, the higher the percentage of ethylene oxide in the copolyetherester, the higher degree of water permeability. Random or block copolymers of ethylene oxide containing minor portions of a second poly(alkylene oxide)glycol can be used. Generally, if a second monomer is present, the second monomer will constitute less than about 30 mol percent of the poly(alkylene oxide)glycols, and usually less than about 20 mol percent. Representative long-chain glycols include poly(ethylene oxide)glycol, ethylene-oxide capped polypropylene oxide glycol, mixtures of poly(ethylene oxide)glycol with other glycols such as ethylene oxide capped poly(propylene oxide)glycols and/or poly(tetramethylene oxide)glycol provided the resulting copolyetherester has an amount of ethylene oxide groups of at least about 25 weight percent. Copolyetheresters prepared from poly(ethylene oxide)glycols having a molecular weight of from about 600 to 1500 are preferred because they provide a combination of superior moisture vapour permeability and limited water swell and, when formed into a film, they exhibit useful properties over a wide temperature range.

20

The term "short-chain ester units" as applied to units in a polymer chain of the copolyetheresters refers to low molecular weight compounds or polymer chain units having molecular weights less than about 550. They are made by reacting a low molecular weight diol or a mixture of diols (MW below about 250) with a dicarboxylic acid to form ester units represented by Formula (II) above.

25

Included among the low molecular weight diols which react to form short-chain ester units suitable for use for preparing copolyetheresters are acyclic, alicyclic and aromatic dihydroxy compounds. Preferred compounds are diols with 2-15 carbon atoms such as ethylene, propylene, isobutylene, tetramethylene, 1,4-pentamethylene, 2,2-dimethyltrimethylene, hexamethylene and decamethylene glycols, dihydroxycyclohexane, cyclohexane dimethanol, resorcinol, hydroquinone, 1,5-dihydroxynaphthalene, etc. Especially preferred diols are aliphatic diols containing 2-8 carbon atoms, most especially

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and where more than one ring is present, they can be joined by aliphatic or aromatic divalent radicals or divalent radicals such as $-O-$ or $-SO_2-$.

Representative aliphatic and cycloaliphatic acids which can be used are sebacic acid, 1,3-cyclohexane dicarboxylic acid, 1,4-cyclohexane dicarboxylic acid, adipic acid, glutaric acid, 4-cyclohexane-1,2-dicarboxylic acid, 2-ethylsuccinic acid, cyclopentanedicarboxylic acid decahydro-1,5-naphthylene dicarboxylic acid, 4,4'-bicyclohexyl dicarboxylic acid, decahydro-2,6-naphthylene dicarboxylic acid, 4,4'-methylenebis(cyclohexyl) carboxylic acid, 3,4-furan dicarboxylic acid. Preferred acids are cyclohexane-dicarboxylic acids and adipic acid.

Representative aromatic dicarboxylic acids include phthalic, terephthalic and isophthalic acids, bibenzoic acid, substituted dicarboxy compounds with two benzene nuclei such as bis(p-carboxyphenyl)methane, p-oxy-1,5-naphthalene dicarboxylic acid, 2,6-naphthalene dicarboxylic acid, 2,7-naphthalene dicarboxylic acid, 4,4'-sulfonyl dibenzoic acid and C_{12} alkyl and ring substitution derivatives thereof, such as halo, alkoxy, and aryl derivatives. Hydroxyl acids such as p-(beta-hydroxyethoxy)benzoic acid can also be used providing an aromatic dicarboxylic acid is also present.

Aromatic dicarboxylic acids are a preferred class for preparing the copolyetherester polymers useful for this invention. Among the aromatic acids, those with 8-16 carbon atoms are preferred, particularly terephthalic acid alone or with a mixture of phthalic and/or isophthalic acids.

The copolyetheresters contain about 25-80 weight percent short-chain ester units corresponding to Formula (II) above, the remainder being long-chain ester units corresponding to Formula (I) above. When the copolyetheresters contain less than about 25 weight percent short-chain ester units, then the crystallisation rate becomes very slow and the copolyetherester is tacky and difficult to handle. When more than about 80 weight percent short-chain ester units are present, then the copolyetheresters generally become too stiff. The copolyetheresters preferably contain about 30-60, preferably about 40-60, weight percent short-chain ester units the remainder being long-chain ester units. In general, as percent short-chain ester units in the copolyetherester are increased, the

Preferably, the copolyetherester elastomers are prepared from esters or mixtures of esters of terephthalic acid and isophthalic acid, 1,4-butanediol and poly(tetramethylene ether)glycol or ethylene oxide-capped polypropylene oxide glycol, or are prepared from esters of terephthalic acid, e.g. dimethylterephthalate, 1,4-butanediol and poly(ethylene oxide)glycol. More preferably, the copolyetherester elastomers are prepared from esters of terephthalic acid, e.g. dimethylterephthalate, 1,4-butanediol and poly(ethylene oxide)glycol.

The dicarboxylic acids or their derivatives and the polymeric glycol are incorporated into the final product in the same molar proportions as are present in the reaction mixture. The amount of low molecular weight diol actually incorporated corresponds to the difference between the moles of diacid and polymeric glycol present in the reaction mixture. When mixtures of low molecular weight diols are employed, the amounts of each diol incorporated is largely a function of the amounts of the diols present, their boiling points, and relative reactivities. The total amount of glycol incorporated is still the difference between moles of diacid and polymeric glycol. The copolyetherester elastomers described herein can be made conveniently by a conventional ester interchange reaction. A preferred procedure involves heating the ester of an aromatic acid, e.g., dimethyl ester of terephthalic acid, with the poly(alkylene oxide)glycol and a molar excess of the low molecular weight diol, 1,4-butanediol, in the presence of a catalyst at 150°-160°C, followed by distilling off methanol formed by the interchange reaction. Heating is continued until methanol evolution is complete. Depending on temperature, catalyst and glycol excess, this polymerization is complete within a few minutes to a few hours. This product results in the preparation of a low molecular weight prepolymer which can be carried to a high molecular weight copolyetherester by the procedure described below. Such prepolymers can also be prepared by a number of alternate esterification or ester interchange processes; for example, the long-chain glycol can be reacted with a high or low molecular weight short-chain ester homopolymer or copolymer in the presence of catalyst until randomization occurs. The short-chain ester homopolymer or copolymer can be prepared by ester interchange from either the dimethyl esters and low molecular weight diols as above, or from the free acids with the diol acetates. Alternatively, the short-chain ester copolymer can be prepared by direct esterification from appropriate acids, anhydrides or acid chlorides, for example, with diols or by other processes such as reaction of the acids

used at temperatures below the softening point of the prepolymer. The major disadvantage is the long time required to reach a given degree of polymerization.

Although the copolyetheresters possess many desirable properties, it is sometimes advisable to stabilize these compositions further against heat or light produced degradation. This is readily achieved by incorporating stabilizers in the copolyetherester compositions. Satisfactory stabilizers preferably comprise phenols, especially hindered phenols and their derivatives, amines and their derivative, especially arylamines.

10 Representative phenol derivatives useful as stabilizers include 4,4',-bis(2,6-di-
 tertiarybutylphenol); 1,3,5-trimethyl-2,4,6-tris[3,5-di-tertiary-butyl-4-
 hydroxybenzyl]benzene and 1,6-bis[3,5-di-tert-butyl-4-
 hydroxyphenyl]propanamido]hexane. Mixtures of hindered phenols with co-stabilizers
 such as diaurylthiodipropionate or phosphites are particularly useful. Improvement in light
 15 stability occurs by the addition of small amounts of pigments or the incorporation of a light
 stabilizer, such as benzotriazole ultraviolet light absorbers. The addition of hindered amine
 photostabilizers, such as bis(1,2,2,6,6-pentamethyl-4-piperidinyl) n-butyl-(3,5-di-tert-
 butyl-4-hydroxybenzyl)malonate, usually in amounts of from 0.05-1.0% by weight of the
 20 copolyetherester, are particularly useful in preparing compositions having resistance to
 photodegradation.

Various conventional fillers can be added to the copolyetheresters usually in amounts of
 from about 1-10 percent by weight of the copolyetherester(s). Fillers such as clay, talc,
 alumina, carbon black and silica can be used, the latter being preferred, and white and light
 25 coloured pigments can be added to the polymers. In general, these additives have the
 effect of increasing the modulus at various elongations.

Example 1

The copolyetherester was extruded through a film-forming die onto a water-cooled rotating quenching drum to yield a cast extrudate. The cast extrudate was heated to a temperature of about 40°C and then stretched longitudinally at a forward draw ratio of 3.0:1. The sheet
5 was passed into a stenter oven, where the sheet was heated to about 80°C and stretched in the sideways direction to approximately 3.0 times its original dimensions. The biaxially stretched sheet was heat-set at a temperature of 160°C. The final thickness of the sheet was 13µm. The sheet was analysed as described herein and demonstrates the following properties:

10	MVTR:	3676g/m ² /day
	Hydrohead:	>1000 cm
	Tensile strength at break:	1.4 daN (MD) 0.7 daN (TD)
	Elongation at break:	111% (MD) 15 220% (TD)
	Tear Strength:	464 mN (MD) 352 mN (TD)

Example 2

- 20 The procedure of Example 1 was repeated except that the film was heated to a temperature of 60°C in the forward draw.

Example 3

- The procedure of Example 1 was repeated except that the forward draw ratio was 1 and the
25 sideways draw ratio was between 3 and 4.2.

Example 4

- The procedure of Example 1 was repeated except that the forward draw ratio was 2.5 and the sideways draw ratio was between 2.5 and 3.

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Example 5

The procedure of Example 1 was repeated except that the forward draw ratio was 2.5, the sideways draw ratio was between 2.5 and 3, and the heat-set temperature was 120°C.

30%, 50%, or 100% elongation, retracting to the original length, drawing again, and retracting again. In addition, samples were drawn to break after two cycles of the 50% hysteresis curves, and tensile strength and percent elongation were measured according to ASTM D638. In each case, the amount of set was determined by calculating the elongation at which the force dropped to zero in the unload curve. The following results were obtained:

Property	Unoriented Film (28 micron)	Oriented Film (45 micron)	Oriented Film (24 micron)
% Set in 20% hysteresis cycles	3	1	1
% Set in 30% hysteresis cycles	6	2.5	2.5
% Set in 50% hysteresis cycles	13	6.5	6
% Set in 100% hysteresis cycles	41	22	22
Tensile Strength at Break (MPa)	14.6	21.0	18.0
Elongation at Break	400%	200%	144%

10 While this invention has been described with respect to what is at present considered to be the preferred embodiments, it is to be understood that the invention is not limited to the disclosed embodiments. To the contrary, the invention is intended to cover various modifications and equivalent arrangements included within the spirit and scope of the appended claims. The scope of the following claims is to be accorded the broadest interpretation so as to encompass all such modifications and equivalent formulations and functions.

D is a divalent radical remaining after removal of hydroxyl groups from a diol having a molecular weight less than about 250;

wherein said copolyetherester(s) contain from about 25 to about 80 weight percent short-chain ester units.

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5. A film according to claim 1 wherein said copolyetheresters(s) have a moisture vapour transmission rate (MVTR) of at least about 2500 gm.mil/m²/24hrs according to ASTM E96-66 (Procedure BW).

10 6. A waterproof moisture vapour permeable fabric comprising a substrate and an oriented polymeric film according to claim 1.

7. A fabric according to claim 6 wherein the substrate is a woven or knit material comprising polyester or nylon.

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8. A fabric according to claim 6 further comprising an adhesive or primer layer or coating between the substrate and copolyetherester-containing film.

9. A fabric according to claim 8 wherein the adhesive or primer is a polyurethane-based adhesive.

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10. A process for the preparation of a fabric according to claim 6 comprising the steps of forming an oriented film comprising one or more copolyetherester(s) by extruding the copolyetherester(s), quenching the extrudate, stretching the quenched extrudate and
25 optionally heat-setting the oriented film, and laminating the oriented film to a pre-formed substrate.

11. An oriented polymeric film comprising one or more copolyetherester(s) wherein the film is oriented in at least one direction and has a set value less than a corresponding
30 unoriented polymeric film.

INTERNATIONAL SEARCH REPORT

Int. lional Application No
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A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08J5/18 B32B27/36 //C08L67:02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C08J C08B D06N C08G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ, COMPENDEX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Y	US 4 493 870 A (VROUENRAETS CORNELIUS M F ET AL) 15 January 1985 (1985-01-15) cited in the application claims 1,3 column 2, line 39 - line 48 column 5, line 16 - column 6, line 47	1-10
Y	US 5 958 567 A (SUGIURA KATSUHIKO ET AL) 28 September 1999 (1999-09-28) claims 1,6,7 example 1	1-10
X	US 4 342 806 A (KUTSCH HOWARD J) 3 August 1982 (1982-08-03) claims 1,4 column 3, line 39 - column 4, line 42 column 5, line 8 - line 10	1,3,4

☒ Further documents are listed in the continuation of box C.
☒ Patent family members are listed in annex.

* Special categories of cited documents:

- * A document defining the general state of the art which is not considered to be of particular relevance
- * E earlier document but published on or after the international filing date
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- * O document referring to an oral disclosure, use, exhibition or other means
- * P document published prior to the international filing date but later than the priority date claimed
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- * Y document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is taken alone in the art.
- * Z document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
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Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5816 Patentkan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Hillebrand, G

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 00/35167

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AN 1997-214270 [20] WPIX

DNN N1997-176660 DNC C1997-069320

TI Lining with functional layer used in robust, lightweight weatherproof coat
- has laminated functional layer contg. microporous membrane based on
PTFE, polyester, polyurethane, co polyether-amide or co polyether-ester of
polyglycol and lower glycol.

DC A14 A23 A25 A83 F07 P21 P73

PA (ALKU) AKZO NOBEL NV

CYC 1

PI **DE 29607822** U1 19970417 (199720)* 21p <--

ADT DE 29607822 U1 DE 1996-29607822 19960430

PRAI DE 1996-29607822 19960430

AN 1997-214270 [20] WPIX

AB DE 29607822 U UPAB: 19970516

The lining, which can be attached to the inside of a coat, has a functional layer. Also claimed is a high-quality weatherproof outer clothing combination of an outer coat, pref. with sleeves, and this lining.

ADVANTAGE - The coat can withstand any weather conditions, is lightweight and robust and is elegant. The functional layer is permeable to water vapour .

Dwg.0/2

